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Project No. 7743 - Special Analytical Methods

MENTHOL ANALYSIS OF PACKAGING MATERIAL

OBJECT:

To provide a method for determining the amount of menthol that has migrated from SALEM SLIM LIGHTS cigarettes into the packaging material of soft and crush-proof box styles.

SUMMARY:

A capillary gas chromatographic method has been developed for the quantitative determination of menthol in packaging materials of SALEM SLIM LIGHTS soft pack and crush-proof box styles.

The analytical procedure consists of extraction of menthol from the packaging material of one pack of cigarettes with methylene chloride. Separation and quantitation of the menthol is performed on a HP 5880A GC equipped with a 30m x 0.312mm DB-1 fused silica capillary column and a flame ionization detector. Anethole is used as the internal standard.

Several determinations of the relative standard deviation for the gas chromatographic procedure resulted in an average value of 8.0%. Menthol recovery was found to be 99%.

The soft pack of SALEM SLIM LIGHTS packaging material was shown to contain 1.0% to 1.5% of total pack menthol for packs stored at room temperature; 2% of total pack menthol was found for soft packs stored at 100° for 30 days as well as 60 days.

The crush-proof box (CPB) style was shown to contain more menthol than the soft pack packaging material. The CPB at room temperature had 3.4% to 3.8% of total pack menthol while those stored at 100° for 30 and 60 days contained 5.0 to 5.6% of total pack menthol.

STATUS:

The method development phase of this project has been completed. Further work will be done as requested by other R&D Divisions.

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I. INTRODUCTION:

At the request of the Integrated Technology Division, a method for determining the amount of menthol migration from the cigarettes to the packaging materials at different temperatures and storage times was developed. It was postulated by the Integrated Technology Division that the CPB style was absorbing menthol from the cigarettes and that a soft pack style might alleviate this problem.

SALEM SLIM LIGHTS products were packaged in the soft pack and crush-proof box style and stored at three different temperatures (40°, 70°, 100°) for different time intervals. The packaging materials for these products were subsequently analyzed for menthol.

II. EXPERIMENTAL

A. Instrumentation

1. HP 5880A CAPILLARY GAS CHROMATOGRAPH - equipped with flame ionization detector and multi-ramp temperature programmer.

2. DB-1 CAPILLARY COLUMN - 30m x 0.312 mm ID fused silica (J & W Scientific)

B. Materials

1. METHYLENE CHLORIDE - (B & J Scientific)

2. INTERNAL STANDARD - anethole (Eastman) 0.1g/liter

3. EXTRACTION SOLUTION - methylene chloride containing 100 µl/liter of anethole

4. MENTHOL - (Fisher Scientific)

5. FILTERS - (Gelman Science Disposable Filter Assembly, 0.45 µm)

6. SYRINGE - 10-cc glass syringe

7. JARS - 16 oz (Smith glass jar with threaded cap)

8. WATCHGLASS - 3 cm diameter

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C. Procedure

1. Sample Preparation

The packaging material from a pack of cigarettes is cut into small pieces approximately 2 cm square and placed in a 16-oz jar. Pipet 100 ml of methylene chloride into the jar making sure all of the material is covered. Pipet 10 ml of the internal standard solution into the jar, stir and place a watchglass on top of the material to keep it submerged in the extracting solution. Cap the jar and allow the mixture to stand for at least 30 minutes with occasional stirring of the contents. Pipet one ml of the solution into the barrel of a 10-cc glass syringe equipped with a Gelman 0.45 µm disposable filter. Push the sample through the syringe into a GC autosampler vial.

2. Calibration of Instrument

A calibration mixture is prepared and analyzed using the same instrumental conditions that will be used for the samples. The 5880A soft-ware package uses the data from these calibration runs to generate response factors which are then saved in a calibration table and used for the sample runs.

3. Chromatographic Conditions

Injection: Splitless injection (split opened 30 seconds after injection)
Column Head Pressure: 30 psig
Injector Temperature: 220°C
Column Oven Temperature: 100°C
Detector Temperature: 300°C
Make-up gas (He) flow rate: 40 ml/min.

III. DISCUSSION:

Figure 1 is a chromatogram of the methylene chloride extract of a SALEM SLIM LIGHTS pack. Peak identification was performed by GC/MS and by GC retention times of authenic menthol and anethole. Baseline resolution was obtained which allowed accurate quantitation of menthol. Table 1 shows the statistics for repetitive runs for each pack style and temperature conditions. From these data an average value of 8.0% was found for the relative standard deviation. Menthol recovery was found to be 99% by standard addition.

It is apparent from these data that there is more retention of menthol by the crush-proof box packaging material than by the soft pack material. However, in both packs the retained menthol is a very small amount of the total pack menthol.

The storage temperature of these cigarettes did not significantly affect the menthol retention for the soft packs. There was somewhat more of an increase in the amount of menthol retention for the crush-proof box style. However, even at elevated temperatures, the amount of retained menthol was not alarming.

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CHROMATOGRAM OF MENTHOL FROM SALEM SLIM LIGHTS
PACKAGING MATERIAL

Figure 1

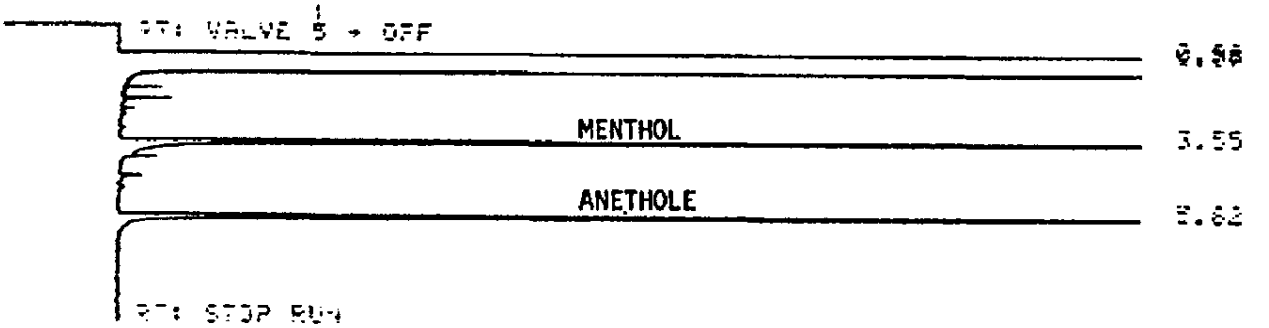

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TABLE 1

MENTHOL CONTENT OF PACKAGING MATERIAL

	SOFT PACK		
	ZERO TIME	30 DAYS/100°F	60 DAYS/100°F
Mean (mg/pack)	0.354	1.59	1.58
Standard deviation	0.040	0.115	0.160
Maximum value	0.397	1.82	2.02
Minimum value	0.319	1.47	1.42
Range	0.078	0.351	0.60
Total pack menthol	78-90 mg/pack	78-90 mg/pack	78-90 mg/pack
% menthol in packaging material	~ 0.5	~ 2.0	~ 2.0

	CRUSH-PROOF BOX		
	ZERO TIME	30 DAYS/100°F	60 DAYS/100°F
Mean (mg/pack)	0.541	4.64	4.15
Standard deviation	0.080	0.163	0.103
Maximum value	0.616	4.85	4.41
Minimum value	0.430	4.34	4.06
Range	0.186	0.503	0.345
Total pack menthol	78-90 mg/pack	78-90 mg/pack	78-90 mg/pack
% menthol in packaging material	~ 0.7	~ 5.0	~ 5.0

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IV. CONCLUSION

A quantitative analytical procedure for menthol retained by packaging material has been developed. The method has been useful in determining small amounts of menthol in packaging material of SALEM SLIM LIGHTS in soft pack and crush-proof boxes stored at different temperatures.

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